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Results of the Third CRC Cooperative Test Program on Hydroperoxide Potential of Jet Fuels

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Eight laboratories conducted tests on nine fuels to evaluate an accelerated storage stability test procedure for measuring the hydroperoxide forming potential of jet fuels. Based on the cooperative work reported herein the 65°C accelerated test readily distinguishes between stable and unstable fuels in approximately 3 weeks stress time. Consequently it appears useful for screening jet fuels for their long-time oxidation stability. Thus this test is recommended as a Go-No-Go test and this is what was sought in these studies. On the other hand, the variability of results within and between laboratories would seem to preclude its use as a precise quantitative					
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RESULTS OF THE THIRD CRC COOPERATIVE TEST PROGRAM ON HYDROPEROXIDE POTENTIAL OF JET FUELS

I. INTRODUCTION

The third cooperative hydroperoxide test program was carried out under Coordinating Research Council (CRC) auspices to investigate further the development of an accelerated test for hydroperoxide potential of jet fuels using for analysis ASTM D3703-85 ("Standard Test Method for Peroxide Number of Aviation Turbine Fuels"). This program was agreed upon and authorized by the CRC Panel on Hydroperoxide Potential at its meeting April 29, 1985 at Dayton, Ohio. This work is of interest especially for hydrotreated fuels and fuels from non-petroleum sources.

In the first test program, which was carried out in 1982 by six laboratories, selected fuels were stored at 100°C for 7 days and analyzed periodically for peroxides. Results showed wide variations between laboratories and between duplicate samples. These results plus subsequent work at NRL at 43°, 65°, and 80°C (1) indicated that an accelerated test at 80° or 100°C is not predictive of behavior at lower temperatures, including ordinary fuel storage conditions.

In the second round robin program (2), conducted in 1984, five fuels were stored at 65°C for 56 days with intermediate sampling times at 1,3,7,14,21 and 35 days. Procedures were tightened in an attempt to reduce excessive variability of results. Unfortunately some of the fuels sought were not obtained and of the five fuels used, four contained antioxidants and in 8 weeks at 65°C did not exceed 0.5 meq/kg of hydroperoxides. Thus this program was unsatisfactory. However, some data were obtained on the variability within laboratories, between duplicate samples of the same fuel, and between labs. Additional work was done by two laboratories on the effect of reaction time and the substitution of Freon 113 (1,1,2-trichloro-1,2,2-trifluoroethane) solvent for carbon tetrachloride. Also some observations on the iodometric titration were collected.

A calibration sample, consisting of stable t-butyl hydroperoxide dissolved in a very stable jet fuel, was included in the second round robin in order to distinguish variability due to the analytical method from that due to the accelerated test. Thus, analysis of this control sample was free from (a) the complexity

of having a mixture of peroxides of different types and (b) changes due to accelerated storage. It appears that there are no known data of record to support the "repeatability (r)" and "reproducibility (R)" (15% and 60% respectively) stated for the ASTM analytical method. Results from the calibration sample provided such data.

In the second round robin, the within-labs spreads (the immediate repeatability, based on 2 sets of quadruplicates) for the calibration standard varied from 0.2 to 15.4% with an average of 6.2%. The corresponding pooled standard deviation and the value of "r" were 3.5% and 9.8% respectively. The lab averages for peroxide number (the reproducibility between labs) varied from 9.20 to 10.51 meg/kg giving a spread of 13%. The corresponding standard deviation was 14.6% and thus "R" was 41%. These relatively low values indicated that the analytical method contributed only a minor component to the variability of the accelerated test results.

II. EXPERIMENTAL

The third test program was planned to enlarge on the previous programs and to ensure that a majority of the sample fuels would develop significant levels of peroxides (more than 1 meq/kg). the latter goal, fuels were selected carefully from a broader range of sources and it was stipulated that they had to have been hydrotreated and contain no anti-oxidant. Samples chosen included JP-5's, Jet A's, blending stocks and a shale JP-4. are identified in Table 1. Ten or 50 gallons of each fuel were obtained and 2/3 gallon each was supplied to the laboratories. Each fuel was stated by the supplier to contain no anti-oxidant. Fuels #5 and #8 were labelled as having no additives. Fuel samples were shipped under argon but were to be aerated before The plan called for nine fuels and eight putting in the oven. laboratories, as compared to the 5x6 matrix in the second round robin, and 3 sample bottles of each fuel instead of 2. This was to permit improved statistical conclusions. The instructions called for putting 400 ml samples in each bottle so that at least 50 ml could be taken at each sampling time. Note that only one analysis per bottle was made at each time. One analysis on each of three identical samples (bottles) at each time is more useful than triplicate analyses on one bottle. Thus each laboratory set up 27 samples instead of 10 in Round Robin 2, but the number of sampling times was 6 instead of 8. It was intended originally to limit the time at 65°C to two or three weeks. However, screening tests at NRL indicated that a longer time was needed in order to develop more peroxides. Thus, the time was extended to 6 weeks with analyses at 0, 7, 14, 21, 28 and 42 days. Greater access of samples to atmospheric oxygen while in the oven was provided in this program. A calibration sample, as described above for the previous program, was furnished again to check the variability due to the analytical method alone (and also biases between laboratories). The hydroperoxide level this time was approximately 3

instead of 10 meq/kg. Instructions and notes on procedure were distributed to participants in a letter dated April 7, 1986 (3). Table 2 lists the laboratories and personnel involved.

III. RESULTS

Analytical results of the accelerated test program are shown in Table 3 together with averages. The averages are graphed in Figs. 1-9. These results should be studied in comparison with the results of the second round robin, which were reported to the Coordinating Research Council October 29, 1984 (2). Note that Texaco conducted duplicate rather than triplicate bottle tests and duPont omitted analysis at 3 weeks. It is apparent that appreciable variation exists between bottles and between laboratories. Development of hydroperoxides in fuels involves free radical reactions among hundreds of compounds and appears to be inherently The components of variability are discussed in the following sections. These include variability of the analytical method, variability between triplicate fuel samples within one laboratory, and variability between laboratories. In spite of such variability it appears, on careful examination of the results, that the 65°C test can distinguish between stable and unstable fuels in about three weeks (see Section F below).

A. Analytical Variability

The non-stressed control sample (see below) provided the primary data on variability attributable to the iodometric analytical method. Additional information was obtained from duplicate fuel analyses. Such duplicate results (same bottle, same day) were reported by Texaco, NIPER and Exxon (see Table 3). (Repeat analyses had been requested whenever a set of triplicate bottles varied by more than 15% or 0.3 meq/kg in the case of results below 1 meq/kg). The close agreement of these 76 pairs of data supports strongly the conclusion that the differences between bottles (see below) are real. An analysis of the data is as follows:

Range of differences between duplicatesa	0-40%
Average difference	5.0%
Pooled standard deviation of individual	
values ^b	6.0%
Repeatability r ^C	16.8%

b p.S.D. =
$$\sqrt{\frac{\text{Ed}^2}{2n}}$$
 where d = % difference and n = no. of pairs (4)

 $c r = 2\sqrt{2} \times std. dev.$

Note that differences and standard deviations must be shown on a percentage rather than an absolute basis because peroxide values vary with fuel and time. The agreement shown by these data represents the repeatability of the inalytical method - as applied to jet fuels. The repeatability "r", as used by ASTM, can be calculated from the standard deviation as shown above. It is defined as "the difference between two successive test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty: 0.15% where X = the average of the two test results." The 16.3% above compares closely with 12.2% for the control sample in this round robin, 9.8% in the second round robin (see below), and 15% stated in ASTM Method D-3703-85 for "r".

Pratt & Whitney and Southwest Research Institute also carried out some repeat analyses 1-4 days later than the originals. Since the peroxide values were changing very rapidly with stress time, it is not possible to compare these paired values. However, when graphed as a function of the number of days at 65°C, the Pratt & Whitney data support the conclusion above that the bottle differences do represent real differences in reaction rates between bottles and are not due to sampling or titration techniques. Furthermore the percent spreads for repeat sets of triplicates were as great as for the initial set. On the other hand, the Southwest Research analyses showed appreciable decreases as well as increases with the additional 1-3 days at 65°C.

B. Variability of Triplicate Fuel Samples (Bottles)

Variations between triplicate bottles as expected were significantly greater than the analytical variability. It is obvious that bottle differences were real. These variations are shown in two ways. In Table 3, variations greater than 1.5 to 1 and greater than 3 to 1 are indicated by superscripts on the averages. Including cases where all three bottles were zero, and excluding a few cases where peroxide numbers were small and of questionable significance, 62% of variations were less than 1.5 to 1, 17% were 1.5-3 to 1 and 13% were greater than 3 to 1. Table 4 shows the percent difference between the highest and lowest of each set of triplicates. These vary from 0 to 302% of mean with an average of 45% (40% is equivalent to 1.5 to 1). The average spread between duplicate bottles in the previous round robin was 18%. This was based on fewer data and is not altogether comparable here. Triplicate variability by either method above is only a little higher (possibly not significantly) for the high peroxide fuels (#4-7) and slightly lower for the low fuels (#1, 2, 3, 8, The values in Table 4 varied significantly between labs as follows (for all fuels and stress times):

Laboratory	Mean	Std. Dev.
NRL	66%	84%
NAPC	68	93
P&W	16	21
duPont	16	22
Texaco	26	22
NIPER	77	82
SWRI	60	77
EXXON	<u>38</u>	<u>57</u>
average	45%	

Note: "Mean" and "Std. Dev." refer to all the values listed in Table 4 for each laboratory.

Also the repeatability "r" (i.e., within sets of triplicates, or within laboratories) was calculated for some representative combinations of fuel and stress time where the peroxide numbers were not zero. Values obtained from the data at four weeks, for example, varied from 20 to 461% of mean with an average of 209%. Note that "r" is calculated as $2\sqrt{2}$ times the pooled standard deviation of triplicate sets, s_w .

C. Variation Between Laboratories

Variations between laboratories were appreciable and greater than the variability of triplicates. Also they were greater than in the second round robin, which produced much less data. Averages for each fuel, time period and laboratory are listed in Table 3 and plotted in Figures 1-9. Some points or labs in the plots appear to be outliers, e.g., NAPC - fuels 2, 3, 8, 9 and Texaco -fuel 5. A rough measure of the interlab variability is the ratio of the highest to the lowest lab using the averages of triplicates. This ratio exceeded 10 in a number of sets and even exceeded 100 in a few cases. In Round Robin 2 the ratios were mostly in the range 3:1 to 10:1. The current data also show that with longer stress times and consequently higher peroxide levels the ratio did not increase. In other words the relative variation between labs was as great at low peroxide levels as at high The reproducibility "R" for all labs was calculated for particular combinations of fuel and stress time. Using all fuels at four weeks, R was 311-909% of mean with an average of 515%. "Mean" is the mean of the eight labs for each fuel.

The data reveal that biases between laboratories do exist, i.e., some laboratories fairly consistently found higher or lower peroxide than others. For example, duPont and Pratt & Whitney were lowest in almost all cases. Texaco and NAPC were highest with five of the fuels and NIPER with three. However, NAPC was high with the low fuels and low with the high fuels while Texaco

and NIPER were high with the high fuels and low with the low fuels. NAPC's high values at 6 weeks may be related to the fact that analyses at 6 weeks (and "C" bottles at 4 weeks) were performed by a different operator. With fuels #6 and #7 there was a sharp division into a high group (Texaco, NIPER, NRL) and a low group (duPont, Pratt & Whitney, NAPC). These results correlate generally with those from the control sample (see below).

Fuels which peroxidized readily were replotted in Figures 10-14 on an expanded scale. With fuels #5-7 the labs found considerable variation in induction time. "Induction time" refers to the time (1-4 weeks here) when the production of peroxides changed from a slow rate to a fast rate. This variable induction period explains much of the variation between laboratories.

D. Control Sample

As indicated above, a control sample was prepared and portions were distributed to the labs to obtain data on the repeatability and reproducibility of the analytical method used in the accelerated test program. The original data are listed in Table 5 with averages and percent range added. Corresponding times for stressed samples are noted only for identification purposes. The control samples were directed to be stored in a refrigerator and no change with time was expected and none was found except in the case of NAPC. Here use of three different operators may have been a factor. The percent range or spread of each set of quadruplicates varied from 2.0% to 17.0% with an average of 7.3%. Compare with the average of 6.2% in Round Robin 2. The pooled standard deviation of individual values (s_w) was 0.0905 or 4.3% of the mean peroxide number of 2.09 meq/kg. This agrees well with the 6.0% shown above for duplicate fuel analyses. The comparable value of s_w in the second round robin was 3.5%. Repeatability "r" for the current data then equals $2\sqrt{2}s_w = 12.23$. The foregoing refers to "immediate repeatability". These values may be compared with the values for duplicate fuel analyses above, which also indicate the immediate repeatability of the analytical method. Calculated values for non-immediate repeatability (i.e., the variation between averages at different times, as 2 and 4 weeks, at the same lab) were 2.2% average range and 1.7% for the pooled standard deviation. a

Reproducibility (the variation between labs) is shown by the following data (extracted from Table 5):

$$\frac{\sqrt{\underline{\mathfrak{r}d^2}}}{\underline{2n}} \times 100$$
grand aver.

Laboratory	Average Peroxide No.
NAPC (2, 6 weeks)	3.33 meg/kg
SWRI	2.41
NIPER	2.35
NRL (25 ml)	2.31
Texaco	2.14
P&W	2.04
DuPont	1.87
EXXON	1.54
Grand average	
(excl. NAPC)	2.09

Statistical values calculated from the data in Table 5 and corresponding values for Round Robin 2 are shown below:

Statistic	Round Robin 3	Round Robin 2
Grand average, meq/kg Spread between averages s_b^{**} Reproducibility R $(2\sqrt{2} s_b)$	2.09 0.87 = 42%* 40.6% 115%	9.91 1.31 = 13%* 14.6% 41%
Aver. spread within labs s_w Repeatability r $(2\sqrt{2} s_w)$	7.3% 4.3% 12.2%	6.2% 3.5% 9.8%

^{* %} of average peroxide number for all labs (2.09 and 9.91)
** Std. dev. between labs. Calculated as shown in reference (4),
p. 32.

Note: NAPC data in Round Robin 3 were excluded.

Obviously the variation between labs is much greater than that within labs. See also the comments above under interlab variation of fuel results concerning laboratories that gave more or less consistently high or low results. The reproducibility R = 115% above is disappointingly high and is to be compared with the 41% found in the second round robin and the 60% stated officially for ASTM Method D-3703. The calculation of R from s_b depends on the definition of R, namely, "The difference between two single and independent results, obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the following values only in one case in twenty: R = 0.60X, where X = the average of two test results."

E. Effect of Sample Size

Some observations on the control sample (Table 5) indicated that peroxide number varies (inversely) with sample size:

Laboratory	Sample Size	Average Peroxide No.
NRL	10 ml (8.06 g) 25 ml (20.16 g) 50 ml (40.3 g)	2.583 2.310 2.060
NIPER	15 g 21 g 23 g 26 g 28 g 31 g	2.473 2.370 2.324 2.311 2.310 2.264

The NRL and NIPER data give a single smooth plot for P.N. vs. sample size. Other laboratories did not report sample size. Unrelated experience at NRL (5) with peroxide determination in fuels has shown no such effect. This effect may explain some of the differences between laboratories.

F. Evaluation of Go/No-Go Test Potential

The military specification for Aviation Turbine Fuel (6) sets a peroxide number maximum of 1.0 meq/kg for JP-5. We therefore examined the Round Robin 3 data to evaluate the P. N. requirement of 1.0 vs the various test times at 65°C.

Table 6 lists the number of laboratories which exceeded the 1.0 limit at the different test times. A high number, 7 or 8, indicates agreement between labs with respect to failure (P.N. >1.0). Note that fuels 4 and 6 were rated as failing in three weeks by all labs (7 of 7) and fuels 5 and 7 failed on most tests (6 of 7). Further, all labs failed these four fuels (4.5.6 & 7) at four weeks. However, two fuels (1 and 9) which showed good stability at three weeks or less, were rated as fails by one lab each at four weeks. At longer times (6 weeks), additional failures were observed.

On the basis of the bulk of the data, fuels 1,2,3,8 and 9 can be classified as satisfactory and fuels 4,5,6 and 7 rated as unacceptable. At stress times of three or four weeks, a peroxide number of 1.0 meg/kg is a good criterion for distinguishing the two sets of fuels. Although other values of the P.N. could be considered to improve the distinction between good and bad fuels, a P. N. of 1.0 is favored on the basis of elastomer tests (7).

Summarizing for the two sets of fuels:

- (a) At 3 weeks, poor fuels exceeded a P.N. of 1.0 in 26 of 28 fuel/lab combinations
- (b) At 3 weeks, good fuels exceeded a P.N. of 1.0 in 0 of 28 fuel/lab combinations
- (c) At 4 weeks, poor fuels exceeded a P.N. of 1.0 in 32 of 32 fuel/lab combinations
- (d) At 4 weeks, good fuels exceeded a P.N. of 1.0 in 2 of 31 fuel/lab combinations.

Data from Round Robin 2 supports the Go/No-Go findings from the current exercise. In the earlier cooperative tests, one fuel was markedly unstable at 65°C and four were classified as stable. All six labs participating in that exercise found more than 1.3 meg/kg of hydroperoxide for the one bad fuel at three weeks also at 2 weeks) and none of the labs found more than 1.0 meg kg for the other four fuels at three weeks. One lab failed one of the acceptable fuels at both 5 and 8 weeks.

IV. CONCLUSIONS

Based on the cooperative work reported herein, the 65°C accelerated test readily distinguishes between stable and unstable fuels in approximately 3 weeks stress time. Consequently it appears useful for screening jet fuels for their long-time oxidation stability. Thus this test is recommended as a GC NO-GC test and this is what was sought in these studies. On the other hand the variability of results within and between laboratories would seem to preclude its use as a precise quantitative tool.

In more detail, the nine fuels examined in this cooperative program can be divided into five acceptable and four unasceptable fuels on the basis of the overall pattern of fuel behavior. Using a criterion of a P.N. of 1.0 meg/kg, six or seven labs out of seven successfully distinguished between the two groups of fuels at three weeks and eight out of eight at four weeks.

Data were obtained on the repeatability r (within laboratories) and reproducibility R (between laboratories) of the analytical method, ASTM D3703, applied to jet fuels by measurements on a non-heat-stressed control sample. The value for r was 12% of mean. This was confirmed by the corresponding value of 17% for 76 pairs of duplicate fuel analyses. On the other hand the repeatability in the accelerated test between triplicate spresses fuel samples within laboratories was 20-461% of mean in selected cases of fuel and stress time. The reproducibility R between laboratories was 272-909% of mean. Some of the latter matter bility was due to laboratory bias, i.e., some laboratory were

consistently high or low. Thus the analytical variability was small while variability between identical stressed fuel samples and between labs was great.

V. REFERENCES

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Table 1 - Test Fuels

Fuel			
No.	Type	Source	Hydrotreatment
1	Jet A	Texaco	Mildly H-treated
2	Blending Stock	Shell, Thornton, UK	Moderately H-processed
3	Blending Stock	Shell, Thornton, UK	Severely H-processed
4		Petro-Canada, Montreal	Hydrotreated
5	Shale JP-4	Wright-Pat. AFB	Hydrotreated
6	Jet A Blending Stock	ESSO Petrol. Corp. Res. Div., Sarnia, Ontario	Hydrofined
7	Jet A Blending Stock	ESSO Petrol. Corp. Res. Div., Sarnia, Ontario	Hydrocracked
8	JP-5	EXXON, Baton Rouge	No H-treatment
9	JP-5	EXXON, Benicia	Mod. H-treatment

Table 2 - Participating Laboratories

1.	NRL	Naval Research Laboratory	Jim Hall
2.	NAPC	Naval Air Propulsion Center	Lynda Turner
3.	P&W	Pratt & Whitney, United Technologies Corporation	Linda Neubauer Paul Warner
4.	duPont	E. I. duPont de Nemours & Co.	Tayman Phillips
5.	TEX	Texaco, Inc.	Salvatore Rand
6.	EXXON	EXXON Research & Engineering Company	William Taylor
7.	SWRI	Southwest Research Institute	Pat Cuellar
8.	NIPER	National Institute for Petroleum and Energy Research	John Goetzinger

Table 3 - Results of 65°C Accelerated Test

	Bottle		Perox	ide Num	ber, me		
LAB	No.	0	1	2	sed at	65°C _4	6
			FUE	L 1 - J	ET A (T	exaco)	
NRL	A B C aver.	.00 .00 .00	.15 .10 .12 .12	.18 .18 .22 .20	.26 .26 .27 .26	.32 .74 .38 .48b	.44 .54 .51 .50
NAPC	A B C aver.	.00 .00 .00	.00 .00 .00	.41 .51 .44	.55 .56 .85 .65	.76 .64 1.51 .97b	1.94 2.03 1.59 1.85
P&W	A B C aver.	.00 .00 .00	.04 .04 .02 .03b	.06 .07 .07	.10 .11 .11 .11	.16 .14 .12 .14	.15 .16 <u>.16</u> .16
duPont	A B C aver.	.00	.03 .04 .04	.10 .09 .09		.16 .09 .12 .12b	.22 .12 <u>.18</u> .17b
TEX	B aver.	.00	.12 .07	.49 .45 .34 .38	.98 .77	2.22 2.31 1.58 1.63	3.70 3.78 2.92 2.87 3.31
NIPER	1 2 3 aver.	.00	.07 .07 .10	.08 .08 .13	.08 .11 .15	.13 .13 .16	.12 .13 .15
SWRI	1 2 3 aver.	.01 <.01 <.01	.30 .43 .20	.27 .56 .37	.55 .14 .52 .40a	.41 .93 .39 .58b	.43 .47 .42
EXXON	A B C aver.	.00	.15 .13 .10 .13	.15 .22 .18	.29 .34 .52 .38b	.28 .27 <u>.32</u> .29	.15 .15 .15

Ratio of highest to lowest bottle >3 b Ratio of highest to lowest bottle 1.5-3

Table 3 (continued)

	Bottle				ber, meq/		
LAB	No.	0	<u>l</u>	2	sed at 65°	4	6
	FUEL	2 -	SHELL, MOD	. PROC	'D BLENDI	NG STOCK	
NRL	A B C aver.	.00 .00 .00	.00 .00 .00	.00 .00 .00	.00 .05 .00	.20 .21 .16 .13	1.03 .76 <u>.99</u>
NAPC	A B C aver.	.00 .00 .00	.00 .00 .00	.00 .00 .00	.00 .00 .00	.49 .00 .00 .16a	4.99 1.20 1.64 2.61a
P&W	A B C aver.	.00	.00 .00 .00	.00 .00 .00	.00 .00 .00	.04 .05 .05	.14 .14 .15
duPont	A B C aver.	.00 .00 .00	.00 .00 .00	.00 .00 .00		.00 .00 .06 .02a	.24 .29 .37 .28
TEX	A B aver.	.00	.00 .00	.00	.00 .02 .01	.10 .14 .12	.56 .78
NIPER	4	.00	.00	.00	.07	. 27	.92
	5	.00	.00	.00	.18	.36	.96
	6 aver.	.00	.00	.19 .06a	.42 .22a	.74 .16b	.96 1.45 1.12b
SWRI		.03 .01 .01	<.01	(.01 (.01 (.01	.64 <.01 <.01 .21a	1.09 .04 <.01 .38a	2.16 .70 <u>.63</u> 1.16a
EXXON	A B C aver.	.00	.00 .00 .00	.01 .00 .00	.00 .00 .00	.00 .00 .00	.27 .14 .12 .18b

a Ratio of highest to lowest bottle >3 b Ratio of highest to lowest bottle 1.5-3

Table 3 (continued)

	Bottle				mber, meq/	kg °C	
LAB	No.	0	l	2	3_	4	6
	FUEL	3 -	SHELL, SEV	. PRO	C'D. BLEND	ING STOCK	•
NRL	A B C aver.	.00	.00 .00 .00	.00	.00 .01 .06 .02a	.08 .07 .11 .09	.21 .40 .48 .36b
NAPC	B C aver.	.00	.00 .00 .00	.00	.00 .00 <u>.17</u> .06a	.33 .18 <u>1.21</u> .57a	.01 .01 1.97 .66a
P&W	B C aver.	.00	.00 .00 .00	.00	.05 .05 .05	.07 .08 .08	.12 .11 .12 .12
duPont	B C aver.	.00	.00 .00 .00	.05 .05 .05		.07 .07 .08	.11 .10 .10
TEX	A	.00	.00	.03	.06	.09	.24
	B aver.	.00	.00	.00 .04 .03	.06	.09	.27
NIPER	7	.00	.00	.00	.00	.04	-11
	8 9 aver.	.00	.00 .04 .01	.00	.00 .00	.00 .08 .04a	.11 .09 .22
SWRI		.01	<.01	<.01 <.01 <.01	<.01 <.01 <.01 <.01	<.01 <.01 <.01 <.01	<.01 <.01 <.01 <.01
EXXON	B C aver.	.00	.00 .00 .00	.00	.07 .00 .00	.05 .06 - <u>.04</u>	.10 .07 .09

a Ratio of highest to lowest bottle >3
b Ratio of highest to lowest bottle 1.5-3

Table 3 (continued)

					er, meq	/kg 5°C	
LAB	Bottle No.	0	1	Stress 2	3	4	6_
			FUEL	4 - PE	TRO-CAN	ADA	
NRL	A B C aver.	.00 .00 .00	1.56 7.32 2.89 3.92a	31.4 64.5 37.2 44.4	103.0 158.0 156.2 139.1b	207.5 257.0 236.4 233.6	361.2 460.8 345.0 389.0
NAPC	A B C aver.	.00	.00 .00 .00	25.1 27.3 24.6 25.7	67.4 50.3 142.2 86.6b	94.6 40.0 288.9 141.1a	297.8 110.7 317.5 242.0b
P&W	A B C aver.	.00	.14 .16 .16 .15	11.9 19.2 17.2 16.1b	96.1 112.2 110.5 106.3	237 286 276 266	471 576 481 509
duPont	A B C aver.	.00 .00 .00	.18 .18 .17	7.45 9.50 8.80 8.58		27.6 38.4 29.1 31.7	46.6 56.5 106.5 69.9b
TEX	A	.00	5.48 5.44	58.1 60.4 63.9	174.8 177.2	237.3 237.6	374.3
	В	•	10.99	88.1 76.3 87.1	254.8 260.1	403.8	409.5
	aver.	.00	8.41b	$\frac{37.1}{72.5}$	216.7	322.0	391.9
NIPER	10	.00	.41	34.3	127.8 129.8	269.6	430.5 427.2
	11	.00	.00	9.9	64.4	241.9	445.6
	12	.00	.51	39.4 43.9	154.9	319.8	459.8
	aver.	.00	.31	28.6a	118.5b	277.1	444.4
SWRI	l 2 3 aver.	.02 <.01 <.01 .01	.95 .93 <u>.97</u>	24.7 30.6 26.0 27.1	97.4	167.7 167.9 160.9 165.5	527.7 643.5 381.1 517.4b
EXXON	A B C aver.	.00	.00 .15 .03	8.70 7.35 4.38 6.81	40.4	65.0 66.2 64.5 65.2	83.0 97.5 75.3 85.3

a Ratio of highest to lowest bottle >3
b Ratio of highest to lowest bottle 1.5-3

Table 3 (continued)

	Dabbla				ber, meg/		
LAB	Bottle No.	_0	week	s Stres 2	sed at 65 3	4	6
UNB	110.			-		_ 	
			FUE	L 5 - S	HALE JP-4		
NRL	A	.00	.06	.11	.91 1.11	8.01	42.1
	В	.00	.06	.08	.57	26.43	107.2
	<u>c</u>	.00	.08 .07	4.65	25.95	63.79	110.7
	aver.	.00	.07	1.62a	9.18a	32.74a	86.7b
NAPC	A	.00	.00	.36	4.09	11.07	106.8
	В	.00	.00	.36	4.60	12.67	10.2
	<u>C</u>	<u>.00</u>	<u>.00</u>	· 42 · 38	$\frac{4.11}{4.33}$	$\frac{27.46}{13.03}$	38.2
	aver.	.00	.00		4.27	17.07b	51.7a
P&W	À	.00	.00	.12	2.6	20.4	97.1
	B	.00	.00	.12	2.3	19.1	110.6
	<u>C</u>	-	<u>.00</u> .00	$\frac{.31}{.18}$ b	$\frac{3.5}{2.8}$ b	$\frac{24.8}{21.4}$	$\frac{138.2}{115.3}$
	aver.				2.0-		
duPont	A	.00	.00	.07		2.07	52.4
	B C	.00	.00	.06 .07		1.89 1.78	44.2
	aver.	.00 .00	<u>.00</u>	.07		$\frac{1.78}{1.91}$	<u>52.8</u> 49.8
					••		
TEX	A	.00	.08	4.54 4.56	28.3 28.4	75.4 74.8	257.5
	В		.18	8.18	35.4	90.9	278.8
			h	8.01	$\frac{35.7}{33.3}$	90.9	260
	aver.	.00	.13b	6.30b	31.9	83.0	268.2
NIPER	13	.00	.00	.00	.03	.12	5.3
	14	0.0	.00	.00	0.6	2.2	5.5
	14	.00	.00	.00	.06	.33	33.9 46.7
	15	.00	.00	.09	.72	13.9	105.8
	aver.	.00	.00	.03a	27a	$\frac{19.9}{5.78a}$	106.1 50.5a
SWRI	1	.06	<.01	.20	1.05	5.88*	60.0
	2 3	<.01	.04	.33	1.08	15.91	50.9
	aver.	$\frac{\langle .01}{.02}a$	$\frac{.07}{.04}a$	29 27b	$\frac{.94}{1.02}$	$\frac{11.82}{13.87}$	$\frac{47.8}{52.9}$
SYVAN	•		22				
EXXON	A	.00	.00	.15	17.2 18.6	48.9 49.7	67.5
	В	.00	.00	.13	4.41	19.7	
			.00	• • •	4.60	18.7	48.8
	С	.00	.05	2.17	14.1	29.3	
					$\frac{24.2}{12.2}$ a	$\frac{28.0}{32.4}$ b	<u>51.5</u> 55.9
	aver.	.00	.02	.82a	12.24	32.45	55.9

KOOO BAXXXXXX BAXXXXII BEERREEN BICK KKKA BEELLEELE BAXAAXX

a Ratio of highest to lowest bottle >3
b Ratio of highest to lowest bottle 1.5-3
* Sample size too large. Omit.

Table 3 (continued)

	Bottle				er, meq/		
LAB	No.	0	1	2_	3	4	6
		FUEL 6	- JET A	BLENDIN	G STOCK,	HYDROFIN	IED
NRL	A B C	.10	.53	1.58	2.71 2.30	5.55 6.66	70.7 89.6
	aver.	.10	1.24 	$\frac{6.41}{3.07a}$	$\frac{30.04}{11.7a}$	81.9 74.0 30.0a	$\frac{297.7}{152.7}$ a
NAPC	A B C aver.	.39 .38 .37	.00 .00 .00	2.69 2.99 <u>2.65</u> 2.78	5.13 4.91 4.90 4.98	5.14 4.66 10.18 6.66b	66.5 51.3 38.2 52.0b
P & W	A B C aver	.08 .09 	.41 .39 .39 .40	.90 .91 <u>.84</u>	1.9 1.9 1.8	3.8 4.2 3.9 4.0	22.2 19.0 19.1 20.1
duPont	A B C aver.	.09 .09 .09	.70 .76 <u>.66</u>	1.80 1.93 1.78 1.84		7.75 6.20 6.75 6.90	23.8 44.5 22.1 30.1b
TEX	A	.08	1.22	6.66 6.62	23.4 23.7	35.1 35.7	139.9 135.8
	B aver.	.09	.98 1.10	4.50 4.56 5.59	16.7 16.7 20.1	46.6 46.7 41.0	177.7 183.2 159.2
NIPER	16	.09	.47	1.04	1.72	3.70	20.1
	17	.09	1.44	3.03	16.08 18.73	63.6 70.6	225.3
	18	.09	.68	2.11	5.99 5.42 8.3a	18.1 17.5 29.5a	196.8 184.4 145.2a
	aver.	.09	.86a	2.06b	8.3ª	29.5a	145.2ª
SWRI	1 2 3 aver.	.20 .01 .11 .14a	1.09 1.18 .94 1.07	1.63 1.86 2.02 1.84	5.54 6.63 6.17 6.11	8.85 10.70 11.33 10.29	88.7 81.9 110.1 93.6
EXXON	A B C aver.	.08 .07 .07	.52 .46 <u>.49</u>	1.11 1.17 1.17 1.15	1.83 1.90 1.77 1.83	$ \begin{array}{r} 3.01 \\ 3.05 \\ \underline{2.73} \\ 2.93 \end{array} $	15.3 14.3 11.7 14.1

a Ratio of highest to lowest bottle >3 b Ratio of highest to lowest bottle 1.5-3

Table 3 (continued)

	204410				er, meq/k ed at 65°		
LAB	Bottle No.	0	1	2	3	4	6
===		FUEL 7	- JET A	BLENDI	NG STOCK,	HYDROCR	ACKED
NRL	A B C aver.	.00	.13 .14 <u>.16</u>	.41 .62 <u>.43</u> .49b	.35 1.63 1.15 1.04a	2.68 3.51 3.64 3.28	102.3 122.2 79.7 101.4b
NAPC	A B C aver.	.00	.00	.17 1.08 .54 .60a	2.38 3.01 1.78 2.39b	4.07 3.77 5.31 4.38	56.4 34.9 12.8 34.7a
P & W	A B C aver.	.00	.04 .04 .04	.21 .15 .20	1.10 .69 .80 .86b	4.2 2.6 3.8 3.5b	39.9 23.4 29.1 30.8b
duPont	A B C aver.	.00 .00 .00	.13 .08 <u>.15</u> .12b	.35 .37 .35		2.75 4.12 3.70 3.52	26.9 29.3 27.0 27.7
TEX	Α	.00	. 28	2.18	6.04	16.1	59.8
	B aver.		.25	2.07 2.70 <u>2.60</u> 2.39	6.07 3.61 8.55 7.32	15.7 24.2 24.7 20.2	59.3 156.2 156.2 107.9b
NIPER	19	.01	.06	.33	1.48	5.05	65.6
	20	.01	.36	2.48	7.52 8.51	21.68	65.2 98.2 96.8
	21	.00	.11	.63	5.67	23.25	161.0
	aver.	.01	-17a	1.22a	6.00 5.11a	16.7ª	$\frac{162.3}{108.2}$ b
SWRI	1 2 3 aver.	.14 .02 <.01 .05a	.77 .44 <u>.42</u> .54b	1.12 1.09 1.20 1.14	4.65 3.72 3.65 4.01	6.63 6.51 9.91 7.68b	63.3 62.0 55.0 60.1
EXXON	A	.00	.00	2.33	6.94	16.6	50.8
	В	.00	.00	2.01	6.23	16.4	50.1
	С	.00	.05	1.16	5.75	17.2	34.2
	aver.	.00	.02a	$\frac{1.61}{1.65}b$	6.31	11.0 14.9a	45.0

a Ratio of highest to lowest bottle >3
b Ratio of highest to lowest bottle 1.5-3

Table 3 (continued)

	Date1.a			de Numb Stress		[/kg 55°C	
LAB	Bottle No.	0	1	2	<u>3</u>	4	6
		FU	EL 8 -	JP-5 (E	XXON, E	BATON ROUGE)
NRL	A B C aver.	.00 .02 .03 .02a	.05 .05 .04 .05	.03 .03 .03	.06 .06 .04 .05	.08 .10 .09	.08 .10 .09
NAPC	A B C aver.	.00	.00	.16 .32 .17 .22b	.00 .00 <u>.39</u> .13a	4.22° .84 .95 .90a	.73 .77 1.12 .87b
P&W	A B C aver.	.00	.03 .02 .04 .03b	.06 .06 .06	.09 .09 .09	.07 .07 <u>.08</u>	.11 .11 .11
duPont	A B C aver.	.00 .00 .00	.00 .00 .00	.00		.03 .00 .00 .01a	.02 .00 .00
TEX	A B aver.	.01 .02 .02b	.07 .05 .06	.14 .09 .12b	.14 .13 .14	.17 .23 .20.	.18 .20 .19
NIPER	22 23 24 aver.	.02 .02 .03 .02	.05 .06 .05	.06 .08 .08	.06 .08 .06	.07 .11 .05	.08 .07 .37
SWRI	1 2 3 aver.	<.01 <.01 <.01 <.01	.20 .18 .12 .17b	.02 .17 .12 .14b	.11 .16 .20 .16b	<.01 .12 .41 .18a	.10 .22 .19
EXXON	A B C aver.	.00 .00 <u>.01</u>	.07 .04 .05	.08 .03 .09	.12 .10 .09	.06 .10 .07 .07b	.08 .07 .09 .08

A Ratio of highest to lowest bottle >3
b Ratio of highest to lowest bottle 1.5-3
c Exclude this outlier.

Table 3 (continued)

	B-5-1	Peroxide Number, meq/kg Weeks Stressed at 65°C								
LAB	Bottle No.	0	weeks l	Stress 2	<u>ed at 65</u>	4	6			
		FU	EL 9	JP-5 (E	XXON, BE	INICIA)				
NRL	A B C aver.	.00 .00 .00	.00 .00 <u>.07</u> .02a	.16 .19 <u>.24</u> .20	.39 .30 .29	.40 .37 <u>.43</u> .40	.79 1.75 .92 1.15			
NAPC	A B C aver.	.00 .00 .00	.00	.00 .00 .39 .13a	.51 .40 .64	1.05 .53 <u>1.70</u> 1.09a	8.72 9.61 1.94 6.76a			
P & W	A B C aver.	.00	.00	.05	.16 .15 .18 .16	.25 .29 .26 .27	.45 .49 .50			
duPont	A B C aver.	.00 .00 .00	.00 .00 .00	.04 .04 .04 .04		.33 .30 <u>.24</u> .29	.47 .49 .53			
TEX	A	.00	.01	.12	.21	.30	.55			
	В	- -	.02	.13	.22	. 4 4	.56 .92			
	aver.	.00	.02b	.13	.22	.37	.94 .74b			
NIPER	25 26 27 aver.	.00	.00	.09 .18 .09	.14 .35 .20 .23b	.18 .49 .30 .32b	.43 .76 .49			
SWRI	A B C aver.	.18 <.01 .03 .07a	.20 .32 .03 .18a	.47 .58 .53	.85 1.02 .29 .72a	.88 1.02 .53 .81b	1.32 1.03 .77 1.04b			
EXXON	A B <u>C</u> aver.	.00 .00 .00	.00	.17 .10 .11 .13b	.34 .23 .16 .24b	.39 .30 .28 .32	.55 .52 .58			

a Ratio of highest to lowest bottle >3
b Ratio of highest to lowest bottle 1.5-3

Table 4 - Variability of Triplicate Fuel Samples

	Perc			Between	Triplica	tes*
LAB	0	<u>l</u>		ssed at 6	5°C4	_6
		,	FUEL	. 1		
NRL NAPC P & W duPont TEX NIPER SWRI EXXON	0 0 0 0 0 0	41 0 61 27 53 38 74 39	21 22 16 11 27 52 73 38	4 46 9 24 52 103 61	88 77 29 57 35 21 93 17	20 24 6 58 25 24 11 53
			FUEL	. 2		
NRL NAPC P & W duPont TEX NIPER SWRI EXXON	0 0 0	0 0 0 0 0 0	0 0 0 0 0 302 0	0 0 159 300 0	26 301 21 33 104 186 0	29 145 7 29 10 46 132 83
			FUEL	. 3		
NRL NAPC P & W duPont TEX NIPER SWRI EXXON	0 0 0 0 0 0	0 0 0 0	0 0 0 0 29 0 0	261 300 0 0 0	44 181 13 13 0	75 296 9 10 12 93 0

^{*} range mean x 100

Note: A range of 1.5:1 = 40% difference. A range of 3:1 = 100% difference.

Table 4 (Continued)

		Perc	ent Dif	ference s Stres	<u>Between</u> sed at 6	Triplica 5°C	tes*
LAB		0	1	_2_	3	4	_6
				FUEL	4		
NRL NAPC P & W duPont TEX NIPER SWRI EXXON		0 0 0 0 0	147 0 13 6 64	75 11 45 24 32 111 22 63	40 106 15 38 81 9	22 176 18 34 52 28 4	30 30 36 37 516
				FUEL	5		
NRL NAPC P & W duPont TEX NIPER SWRI EXXON		0 0 0 0	37 0 0 0 77 0	280 16 100 15 57 48 249	276 12 43 23 256 14 110	171 10 27 15 19 291 30 93	68 1835 89 1933 1933
				FUEL	6		
NRL NAPC P & W duPont TEX NIPER SWRI EXXON		0 5 12 0 12 0 173	90 0 5 14 22 113 22 121	202 12 10 8 38 97 21	233 5 5 24 189 18	141 310 111 111 111 111 111 111 111 111 11	1 + 6 + 8 + 0 0 0
	x 100 range of range of						
				2 3			

Table 4 (Continued)

	Per	rcent D	ifferen	ce Betwe	en Tripli	cates*							
		Week	s Stres	sed at 6	5 °C								
<u>LAB</u>	0	1	2	3	4	_6							
				-									
	FUEL 7												
NRL	0	21	43	123	29	42							
NAPC	0	0	152	51	35	126							
PEW	0	0	32	48	45	54							
duPont	0	58	6		39	9							
TEX	0	11	22	34	43	90							
NIPER	0	167	195	127	109	89							
SWRI	280	65	10	25	44	14							
EXXON	0	300	64	19	42	37							
			FUEL	8									
			FOEL	0									
NRL		31	22	40	20	20							
NAPC	0	0	74	300	169	45							
P&W	0	67	0	0	13	0							
duPont	0	0	0										
TEX		33	43	7	30	11							
NIPER		25	18	40	50	17							
SWRI	0	47	107	57	228	71							
EXXON	0	60	86	27	57	25							
			FUEL	9									
NRL	0		39	30	15	8 3							
NAPC	ä	0	300	46	107	113							
P&W	Ö	Ō	23	18	15	8							
duPont	ā	Ō	0	-	31	12							
TEX	Ō	67	8	5	38	49							
NIPER	0	0	82	92	92	5 3							
SWRI	245	161	21	115	60	5.3							
EXXON	0	0	54	75	34	11							

Note: A range of 1.5:1 = 40% difference. A range of 3:1 = 100% difference.

^{*} range x 100

Table 5 - Control Sample Results Peroxide Number, meq/kg

1. NRL

3 We	eks	5	Weeks	
25 ml	10 ml	25 ml	10 ml	50 ml
2.350	2.633	2.254	2.489	2.060
2.339	2.519	2.248	2.690	
2.315		2.314		
2.431		2.233		
2.364		2.246		
2.360*	2.576	2.259	2.590	2.060
4.9%*	4.4%	3.6%	7.8%	

2. NAPC

	4.9%* 4.4%	3.6% 7.8	
(In the state of t	APC		
	2 Weeks 3.72 3.52 3.78	6 Weeks 2.94 3.03	9 Weeks 1.90 2.10 1.91
3. P8	3.67 7.1%	2.99 3.0%	1.97 9.6%
3. P8	. ·		
	1 Week 2.02 2.03 2.13 2.02 2.05 5.4%	3 Weeks 2.05 2.01 2.02 2.05 2.03 2.0%	
4. di	1Pont		
4. du	6 Weeks 1.84 1.89 1.87 2.7%		
	2 Weeks	4 Weeks	
5. Te	2.13 2.09 2.16 2.10 2.12 3.3%	2.17 2.21 2.03 2.23 2.16 9.3%	
		25	!

<u>l Week</u>	3 Weeks
2.02	2.05
2.03	2.01
2.13	2.02
2.02	2.05
2.05	$\frac{2.03}{}$
5.4%	2.0%

6		W	е	e	k	٤
_	1	•	8	4		
	1	•	8	9		
	ī	•	8	7		
		2		7	ષ્ઠ	

2 Weeks	4 Weeks
2.13	2.17
2.09	2.21
2.16	2.03
2.10	2.23
2.12	$\frac{2.16}{}$
3.3%	0, 7%

Table 5 (continued)

6. NIPER

2 Weeks	4 Weeks
2.310	2.331
2.425	2.287
2.334	2.353
2.473	2.324
	2.264
2.386	2.312
6.8%	3.8%

7. SWRI

2 Weeks	4 Weeks
2.25	2.30
2.54	2.23
2.50	2.60
2.63	2.22
2.48	2.34
15.3%	16.2%

8. EXXON

Initial (8/27/86)	2 Weeks
1.6783	1.5107
1.5844	1.2807
1.6635	1.3701
1.7252	1.5181
1.6629	$\frac{1.4199}{1.4199}$
8.5%	17.0%

^{*} The first value is the average and the second is the ratio of the range to the average as a percent.

Table 6 - Accelerated Test Time Needed to Distinguish

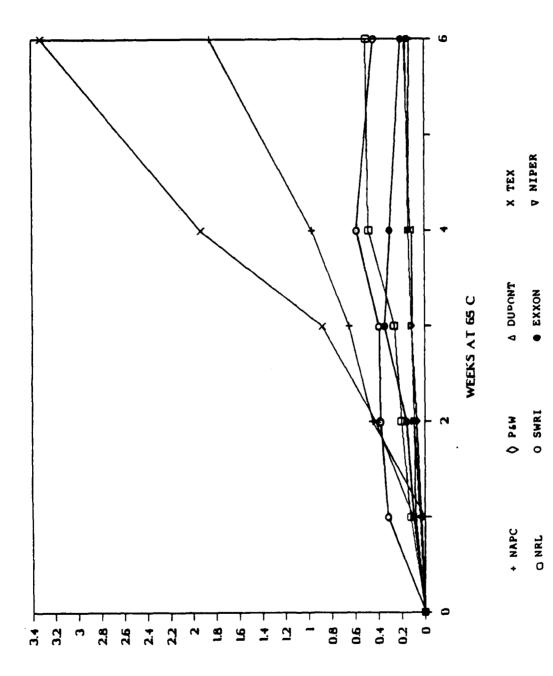
Between Stable and Unstable Jet Fuels

	No.	No. of Labs With Peroxide Numbe Greater Than 1 meg/kg				
		Week	s Stress	ed at 65°	C	
Fuel No.	1	2	<u>3*</u>	_4_	_6_	
1	0	0	0	1	2	
2	0	0	0	0	3	
3	0	0	0	O	0	
4	2	8	7	8	8	
5	0	2	6	8	8	
6	2	7	7	8	8	
7	0	4	6	8	8	
8	0	0	0	0	0	
9	. 0	0	0	1	3	

^{*7} labs reported instead of 8.

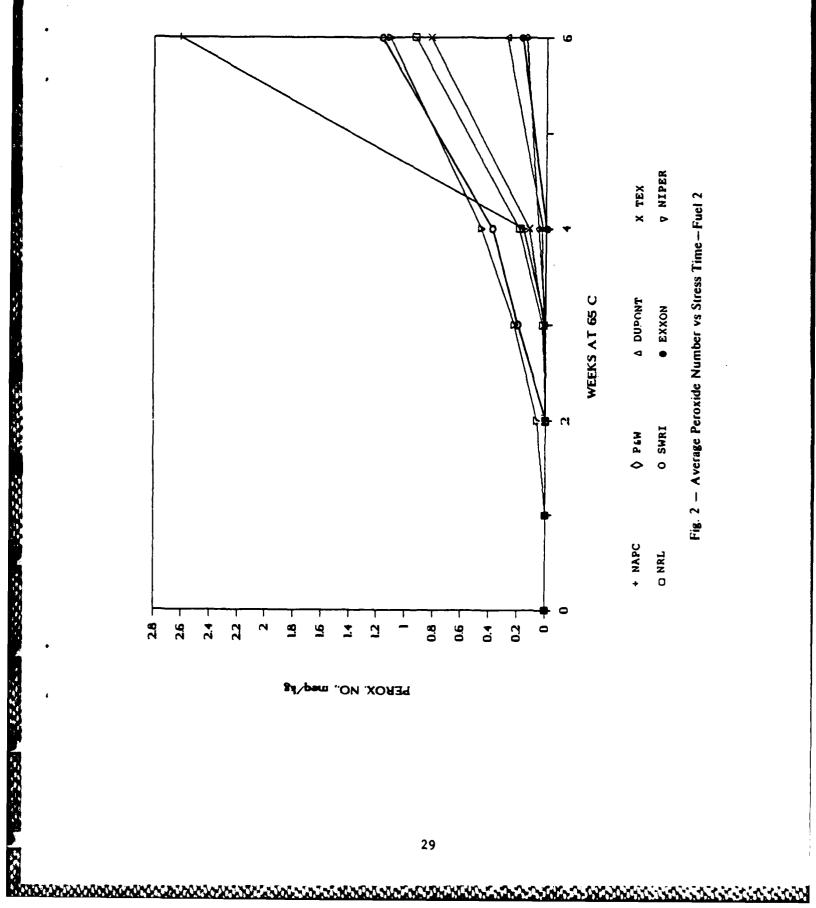
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Note: Fuels #1,2,3,8,9 are classified as stable fuels and #4-7 as unstable fuels.



PEROX. NO. meq / ltg

Fig. 1 - Average Peroxide Number vs Stress Time-Fuel I



V NIPER Fig. 2 - Average Peroxide Number vs Stress Time-Fuel 2 • EXXON

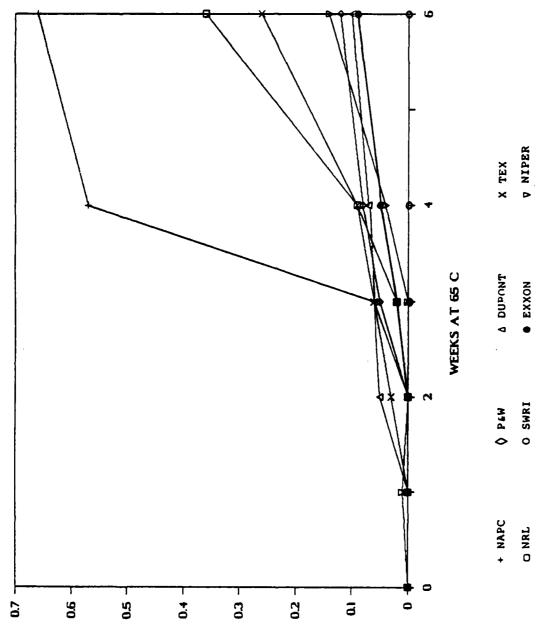


Fig. 3 — Average Peroxide Number vs Stress Time—Fuel 3

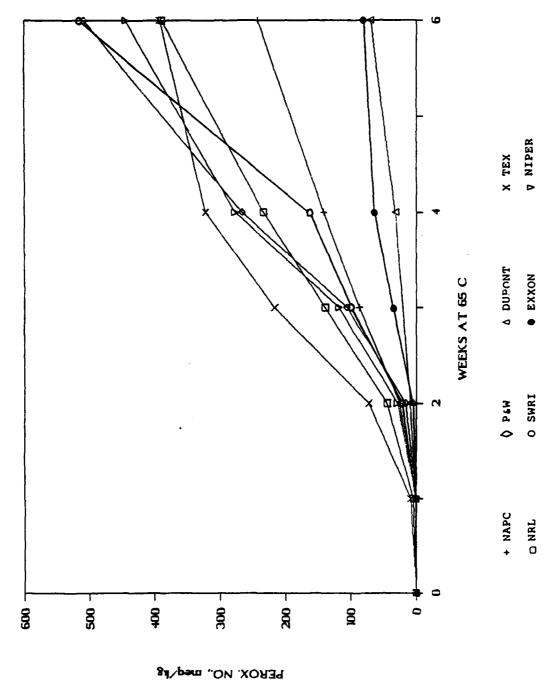


Fig. 4 — Average Peroxide Number vs Stress Time—Fuel 4

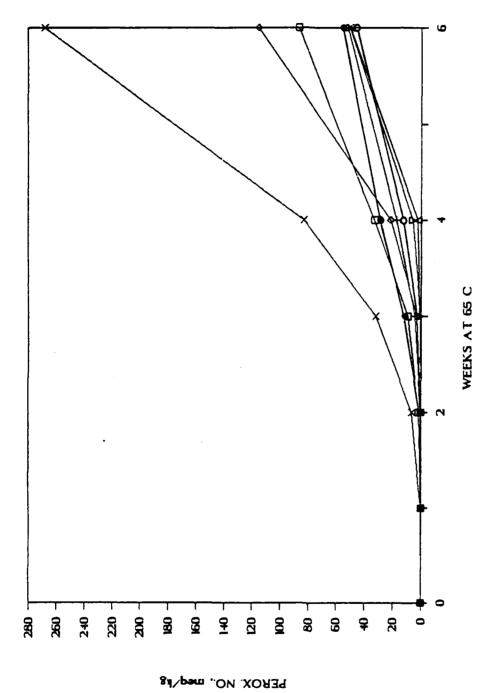




Fig. 5 - Average Peroxide Number vs Stress Time-Fuel 5

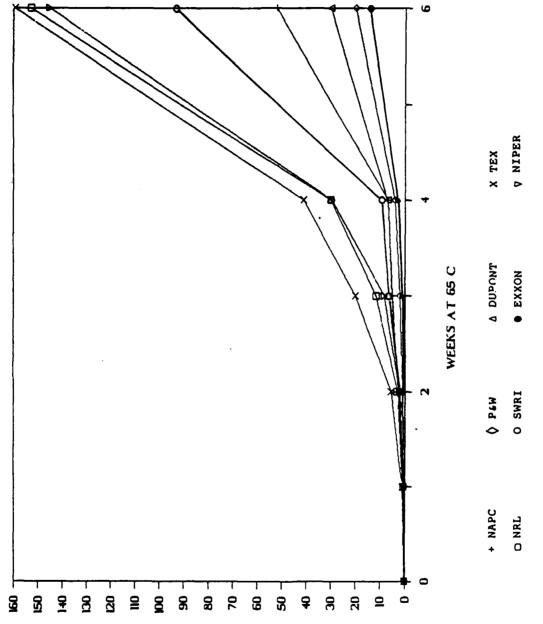
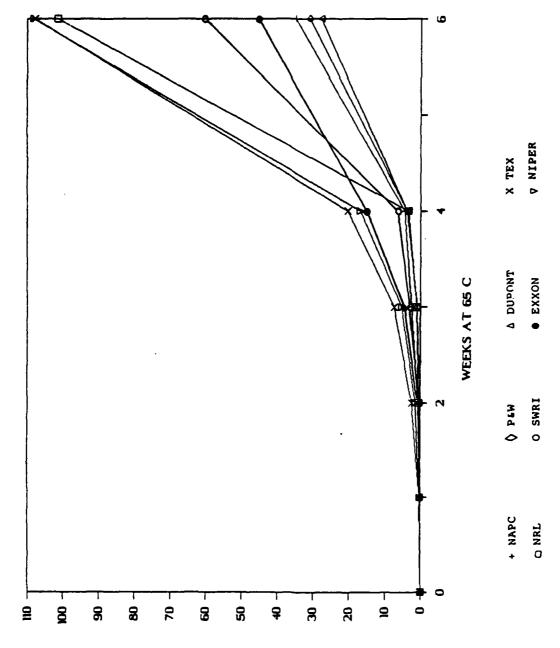
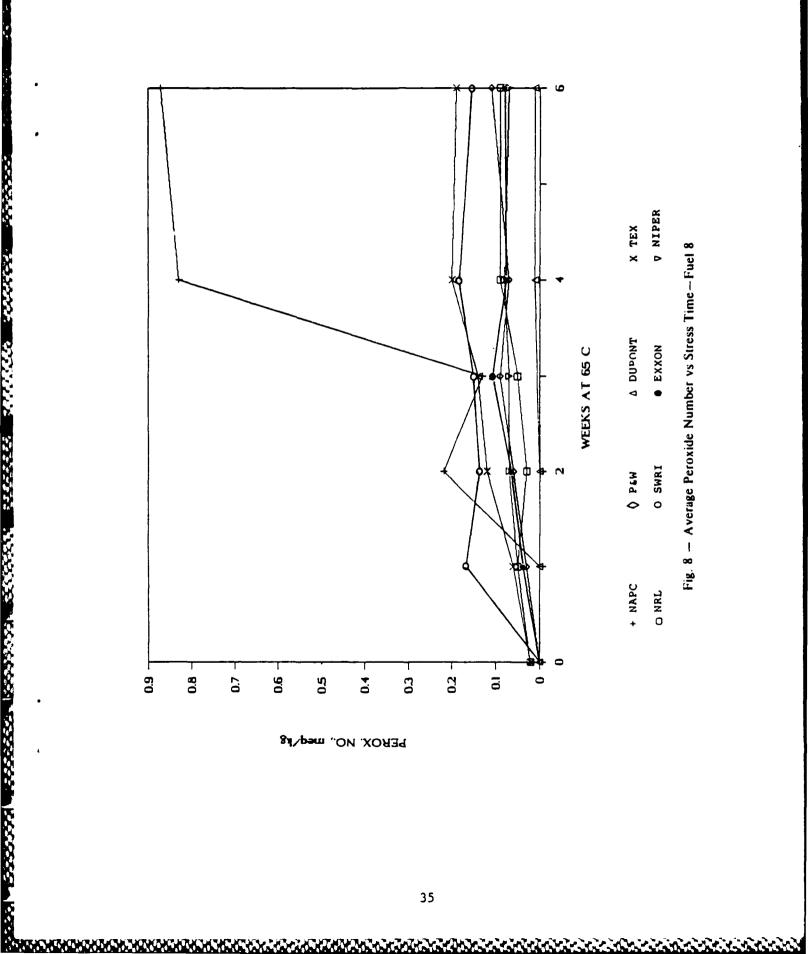
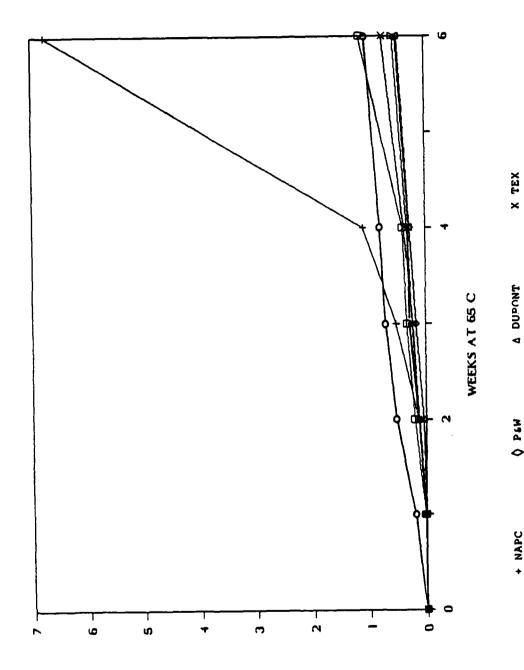


Fig. 6 - Average Peroxide Number vs Stress Time-Fuel 6







PEROX. NO., meq/kg

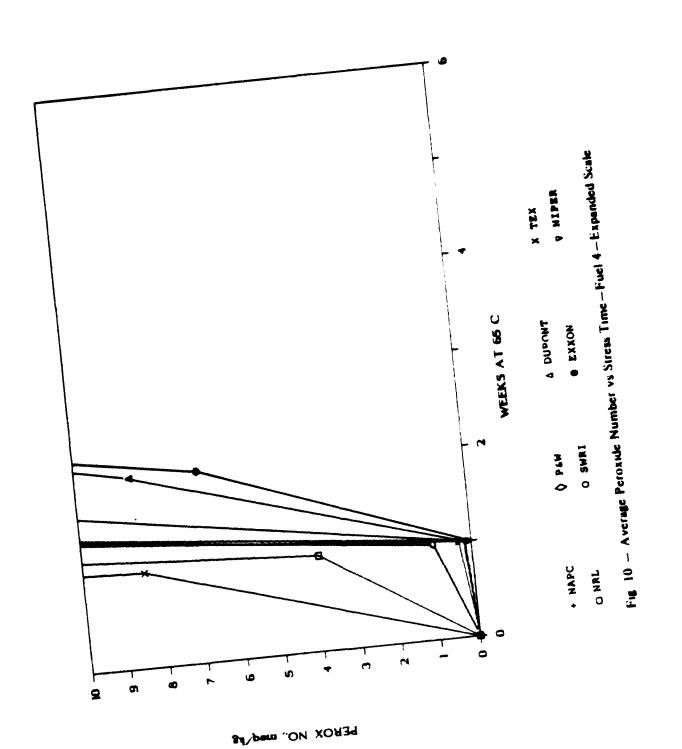
Fig. 9 - Average Peroxide Number vs Stress Time-Fuel 9

V NIPER

• EXXON

O SWRI

+ NAPC O NRL



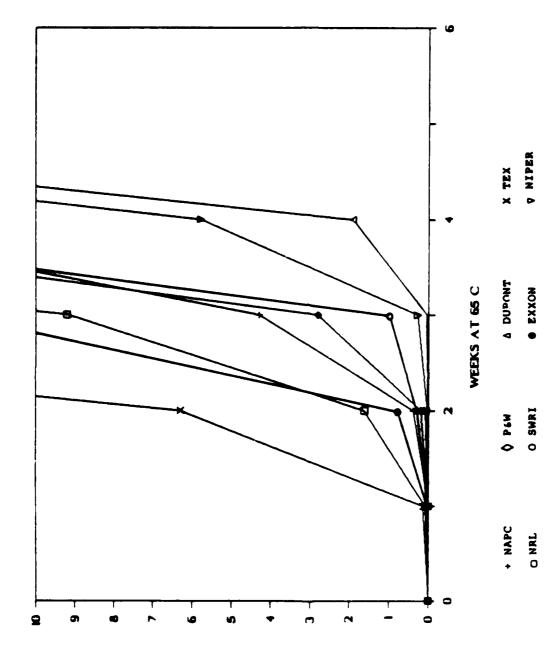


Fig. 11 - Average Peroxide Number vs Stress Time-Fuel 5-Expanded Scale

Change processes and the control of processes.

PEROX. NO., maq./lg

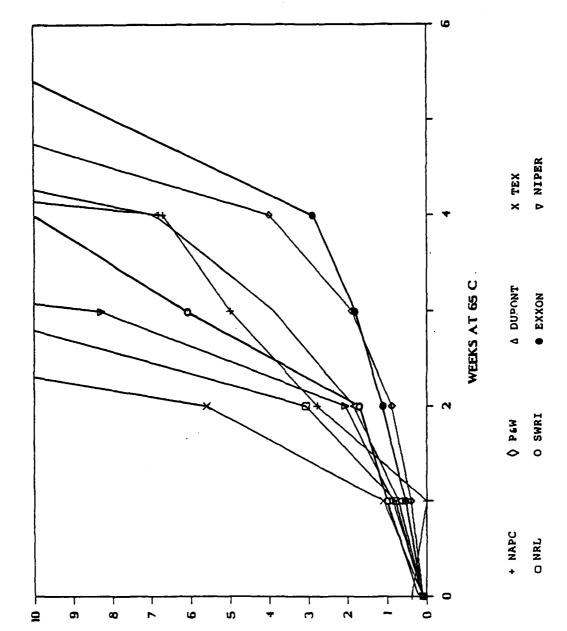


Fig. 12 - Average Peroxide Number vs Stress Time-Fuel 6-Expanded Scale

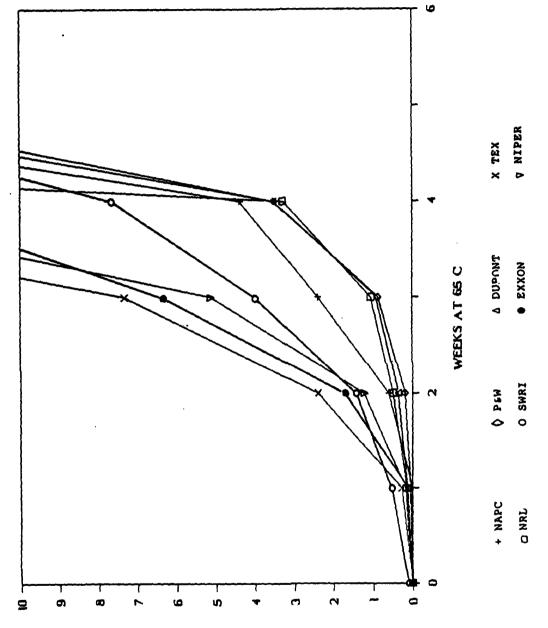
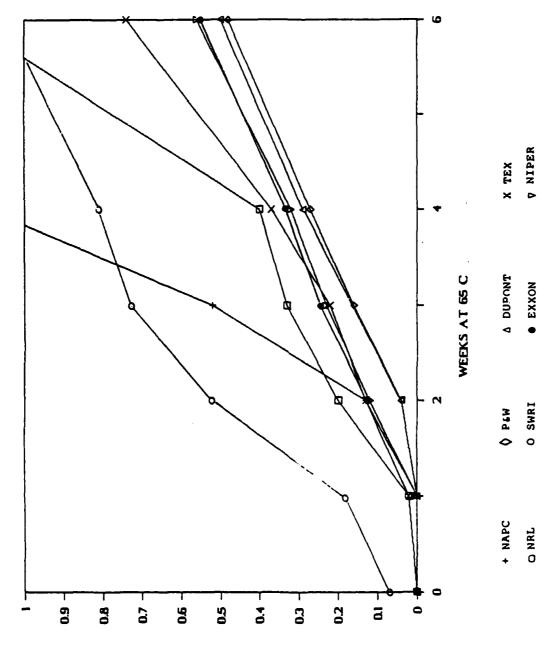


Fig. 13 — Average Peroxide Number vs Stress Time—Fuel 7—Expanded Scale



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Fig. 14 - Average Peroxide Number vs Stress Time-Fuel 9-Expanded Scale